PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Process for the Preparation of Phloroglucinol

We FISONS INDUSTRIAL CHEMICALS LIMITED, formerly Whiffen & Sons Limited, a Britsih Company, of Willows Works, Derby Road, Loughborough, Leicestershire, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the preparation of phloroglucinol.

It has been found that phloroglucinol can be prepared by hydrogenating trinitrobenzene or trinitrobenzoic acid in the presence of solvents and subsequently hydrolysing the product.

Accordingly the present invention provides a process for preparing phloroglucinol which comprises hydrogenating trinitrobenzene or trinitrobenzoic acid, preferably under pressure, in a solution in a solvent containing at least 10% by weight of the trinitrobenzene or trinitrobenzoic acid and in the presence of a hydrogenation catalyst at a temperature of at least 40°C., thereafter separating the catalyst and the solvent from the hydrogenated mixture, hydrolysing the hydrogenated mixture with acid and recovering phloroglucinol.

Example of solvents which may be used in the process of the present invention include ketones such as acetone and methyl ethyl ketone, alcohols such as methyl and ethyl alcohol, esters such as methyl and ethyl acetate and ethers such as dioxane.

The catalyst is preferably that product which is known as "Raney" nickel which is obtained by treating a nickel-aluminium alloy with caustic soda solution. Other catalysts include platinum, palladium or platinum oxide.

The amount of catalyst in relation to the amount of trinitrobenzene compound governs to some extent the temperature at which the reduction is conducted. With a large amount of catalyst present such that the weight ratio of trinitrobenzene to wet catalyst is 2:1 the temperature of reduction is in the range 45—55°C whereas with a small amount of catalyst present such that the weight ratio of trinitrobenzene compound to wet catalyst is 8:1, the temperature of reduction is of the order of 100°C. In general the weight ratio of the trinitrobenzene or trinitrobenzoic acid to wet catalyst is preferably in the range 2:1 to 8:1 and the temperature at which the reduction is carried out is preferably in the range 45°C to 110°C.

Since the hydrogenation involves three phases namely gaseous hydrogen, a solution of nitrobenzene or trinitrobenzoic acid and solid catalyst it is highly desirable if complete reduction is to be obtained, to ensure that mixing of the three phases is efficient.

The acid used in the hydrolysis step of the process of the present invention is preferably a mineral acid such as hydrochloric acid, sulphuric acid, phosphoric acid and the like. Preferably the hydrolysis step is performed at a temperature of 80°C to 105°C.

The following examples in which parts are by weight are given to illustrate the process of the present invention.

EXAMPLE 1.

A solution of 106.5 parts of trinitrobenzene in 1,000 parts of acetone was reduced with hydrogen in the presence of 15 parts of wet Raney nickel catalyst, in a hydrogenator at a pressure of 60 pounds per square inch and a maximum temperature of 85°C. The resulting reduced solution was drawn off from the catalyst and acidified with sufficient hydrochloric acid solution to hydrolyse the tri-

aminobenzene formed in the reduction. After recovery of the acetone by distillation, to raise the boiling point of the triaminobenzene hydrochloric solution to 100°C., the hydrolysis was completed by boiling for at least 18 hours. The resulting solution contained 69 parts of phloroglucinol, equivalent to 85% of the theoretical yield. Solid phloroglucinol may be obtained by concentration and crystallisation of the solution followed by filtering off the product, and drying in a low temperature oven.

EXAMPLE 2.

A solution of 143 parts of trinitrobenzoic acid in 1,430 parts of acetone was reduced with hydrogen in the presence of 50 parts of wet Raney nickel catalyst, in a hydrogenator at a pressure of 60 pounds per square inch and a maximum temperature of 65°C. The resulting reduced solution was drawn off from the catalyst into sufficient hydrochloric acid to hydrolyse the triaminobenzoic acid formed in the reduction. Following the same procedure as in Example 1, the boiling point of the solution was raised to 100°C by recovery of the acetone by distillation. Hydrolysis and decarboxylation was completed by refluxing for 18 hours. The resulting solution, contained 59 parts of phloroglucinol, equivalent to 65% of the theoretical yield. Solid phloroglucinol was recovered by evaporation and crystallisation.

WHAT WE CLAIM IS:—

1. A process for preparing phloroglucinol which comprises hydrogenating trinitrobenzene or trinitrobenzoic acid, preferably under pressure, in a solution in a solvent containing at least 10% by weight of the trinitrobenzene compound and in the presence of a hydrogenation catalyst at a temperature of at least 40°C., thereafter separating the catalyst and the solvent from the hydrogenated mixture, hydrolysing the hydrogenated mixture with acid and recovering phloroglucinol.

2. A process as claimed in claim 1 wherein the solvent is a ketone or an alcohol.

3. A process as claimed in claim 2 wherein the ketone is acetone or methyl ethyl ketone.

4. A process as claimed in claim 2 wherein the alcohol is ethyl alcohol.

5. A process as claimed in any of the preceding claims wherein the catalyst is "Raney" nickel, platinum, palladium or platinum oxide.

6. A process as claimed in any of the preceding claims wherein the weight ratio of the trinitrobenzene compound to wet catalyst is in the range of 2:1 to 8:1 and the temperature at which the reduction is carried out is in the range 45°C to 110°C.

7. A process as claimed in any of the preceding claims wherein the acid used in the hydrolysis step is mineral acid such as hydrochloric acid, sulphuric acid or phosphoric acid.

8. A process as claimed in any of the preceding claims wherein the hydrolysis step is performed at a temperature of 80°C to 105°C.

9. A process for preparing phloroglucinol substantially as hereinbefore described with reference to the preceding examples.

10. Phloroglucinol whenever prepared by the process as claimed in any of the preceding claims.

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